Effect of Pigment Concentration on Mechanical Properties and on color stability of Polycarbonate

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Abstract: - This experimental study deals with the influence of pigments on the mechanical properties of polymeric polycarbonate material Lexan 923c, which is commonly used in industry and is being processed with injection technology. This material was stained with three different pigments from Lifokolor. It was also stained with 6 different concentrations of selected pigments. Emphasis was placed on measuring the tensile strength, impact strength and hardness of polymer mixtures. It also deals with the influence of pigment concentration on colorimetric properties and the concentration of pigment value at which the material needs to be colored. Colorimetric properties and reflective spectra of different concentrations were also measured.

Key-Words: - Injection molding, mechanical properties, colorimetric properties, polycarbonate, pigment concentration, CIE diagram

1 Introduction

Nowadays polymeric materials are irreplaceable in many industries. They are exclusive material for their mechanical properties and shaping capabilities. To obtain higher effectivity of production, a number of complicated parts is made by injection technology. Often, customer requirements are too high. Above all, the biggest problem is adhering to the desired color range and the mechanical properties of the part. Therefore, compromise between materials and pigments should be found.

There are several basic color methods. However, these methods are associated with the final process. We are focusing on the injection process, which has a high ability to reproduce complicated parts.

The granulate for injection can be colored in a dry manner. This is a direct mixing of the natural granulate with the pigments in the mixing device. Thereafter, this prepared mixture is transferred in the hopper of the injection moulding machine. The distribution of the colored concentrate of this mixture is not perfect. Even further strained and sedimentation of the colored concentrate is observed due to the shock of the injection unit. Therefore, this method is suitable for products with lower demands on color accuracy. To obtain more precise distribution of color concentrate, bulk or mass dispensers are used.

The colored concentrate consists of a polymeric carrier which is miscible with the polymer. In the polymeric carrier, the pigment is incorporated up to 90%. The final colour effect must be consist with the customer's requirements. The way of dosing and mixing of the coloured concentrate is important for desired values of the colour shade. The way of dosing is also very important for the final mechanical properties of the part

2 Methods

2.1 Type of polymeric material and pigment

Lexan 923 polycarbonate (PC) from SABIC was selected for studying of mechanical properties. This material is characterized by high impact resistance, it is strong and flexible. Excludes the possibility of cracks. In contact with fire, products become soft and melt, fire is not support and it is not expand. Low weight with good mechanical properties guarantee a long service life. The test specimens were made by injecting this polymeric material with a different concentration of three kinds of pigment colors.

For the coloring of the natural polymeric material, three color pigment types have been used, which are commonly used in industry. These pigments were purchased from LIFOCOLOR s.r.o. in basic color shades. This pigment is stable up to 290 °C

2.2 Formation of the mixture

Dry blending was used for preparing of the mixture. Stirring was always carried out in the exact ratio of Lexan 923 polymer granulate with a certain type of pigment. Mixing systems commonly used in industry were used. The mixtures were mixed in concentrations (1-6)% ; total weight of the final mix was 1500g. The precision laboratory height of the HELAGO HL - 2000i was used for weighing.

Table 1. Formation of the mixture.

$C_{pigment}$	$C_{polymer}$	$m_{pigment}$	$m_{polymer}$	M_{mix}
1%	99%	15g	1485g	1500g
2%	98%	30g	1470g	1500g
3%	97%	45g	1455g	1500g
4%	96%	60g	1440g	1500g
5%	95%	75g	1425g	1500g
6%	94%	90g	1410g	1500g

2.2 Production of test specimens

The test samples were created by injection molding technology on the ARBURG Allrounder 470H 1000-400 Injector. This machine can be optimally dimensioned for applications of all known injection technologies.

Fig. 1 Injection machine

Two types of standardized test bodies were selected. The first body is designed for tensile testing according to ČSN EN ISO 527-1 type 2 and the second body for impact strength is degined according to ČSN EN ISO 179-1

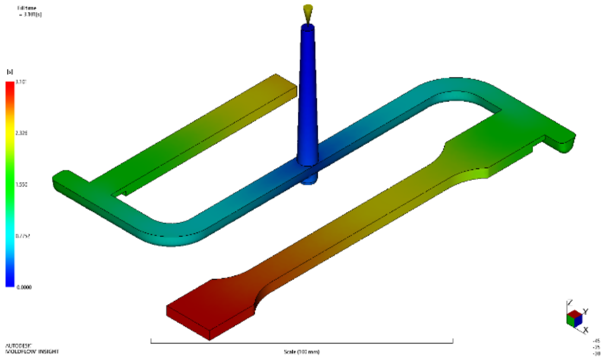

Fig. 2 Injection molded bodies

Table 2. Injection Parameters.

mold temperature	60	°C
plastication pach	24	Mm
injection pressure	800	Bar
touch pressure	300	Bar
time touch pressure	15,5	S
injection speed	40	mm/s
patch	8	mm
cool time	30	S
1.zone temperature	40	°C
2. zone temperature	270	°C
3. zone temperature	275	°C
4. zone temperature	280	°C
5. zone temperature	285	°C
nozzle temperature	290	°C

2.2 Colorimetry

Colorimetry is the optical method based on comparing the intensity of the colored mixture to a non-concentration with a mixture of the same substance of known concentration. The intensity of the characteristic color of the substance depends on its concentration in the mixture. From the physical point of view, the color is determined by the spectral composition by the source of transmitted light. The description of the color scheme is based on the relative intensity of light reflected by the surface of the body or the protruding body.

For the sophisticated assessment of the color is necessary to define three elements : light source, observed object and observer.

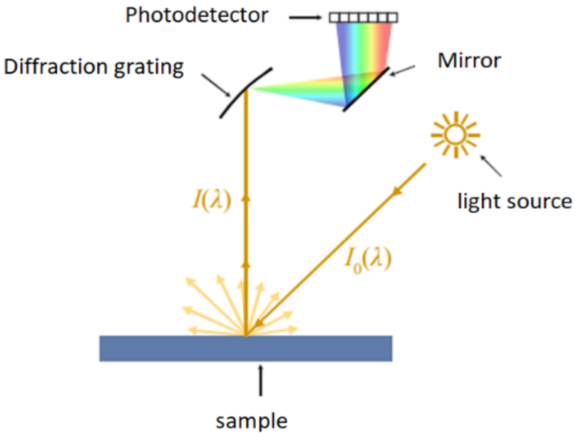

Fig. 3 Color sensing

Reflection tells us how much the object reflects the light of a given wavelength. When we find this parameter for all wavelengths in the range of approximately 380-750 nm, we get the reflective spectrum characterizing the object as show.

$$R_{(\lambda)} = \frac{I_{(\lambda)}}{I_{0(\lambda)}}$$

2.2 Color measurement

Each visible source radiates in the visible area on each wavelength at different intensities. Light from different sources has different colors. Colorimetry attempts to describe the color using continuous functions, which can then be compared to each other.

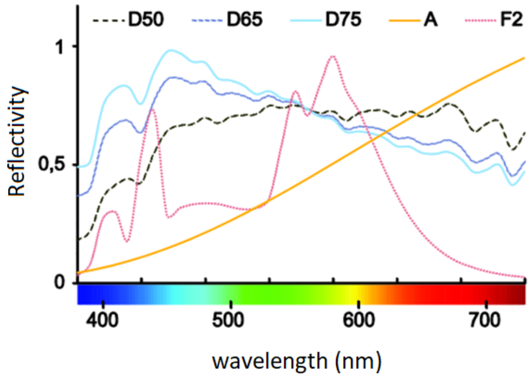

Fig. 4 . Reflective spectrum

2.2 Colorimetric parameters

Measured values are converted using the composite functions to the CIE L* a* b* color space coordinates. Where the parameter L* is a specific light parameter (determinating a light or dark color), on the axis a* are colors from green to red and on b* the colors are from blue to yellow. All colors that we are able to perceive as observers are able to assign specific coordinates in this space. If we want to compare two colors, we can do it by calculating the distance in this space. This distance is called the color deviation ΔE and can be calculated using the equation:

$$\Delta E^*_{ab} = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2}$$

From the coordinates a* and b* is possible to calculate additional color attributes, namely chrom and hue. Chroma C* indicates the color gamut, and increases the distance from the L* axis. The h* is determined by an angle.

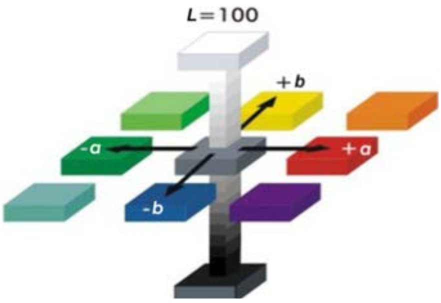

Fig. 5 Color space

3 Methods

Materials were tested by tensile test, flexural impact test, hardness test, and dimensional measurements to evaluate shrinkage. To compare the physical (mechanical) properties of polymer materials (PC) which was influenced by different pigment concentrations, a tensile test machine, charpy hammer, portal gauge and hardness hardness tester (Shore D) was used.

3.1 Impact bending test

The HIT impact hammer automatically detects the used pendulum. Therefore, HIT provides accurate measurement data within the specified range and corresponding to the standard. A double carbon rod was used to make the pendulums. High impact strength and high mass concentration at the point of impact are achieved. The impact hammer has an optional pendulum brake. The electronics include a high-resolution digital sensor for accurate angular swing measurement. Each set of measurements was performed on 20 test bodies

Fig. 6 Impact bending test

3.2 Tensile stress machine

After producing of the testing samples a test was carried out to determine the tensile stress-strain properties and also the test to determine the tear strength. In both cases the testing samples were clamped into jaws at both ends in the tensile stress machine Tensometer 2000 by Alpha Technologies. (Fig. 5). Test sample dumbbell was stretched by the prescribed constant speed 50 mm/min until they were torn. In case of test sample crescent, graves and trouser, stretching speed was 100 mm/min.

Fig. 7 Tensile stress experiment

3.3 Color measurement spectrophotometer

The D65 illumination source is calibrated in the ultraviolet region for the accurate measurement of whitening agents. UltraScan PRO has an extended wavelength range into the near infrared and near ultraviolet that permits the measurement of camouflage materials and UV blockers. The system uses diffuse/8° geometry with automated specular component inclusion/exclusion. It also features three sizes of sample measurement areas with automated lens change. Use UltraScan PRO for both research and quality control. From opaque solids to clear liquids to transparent films, UltraScan PRO precisely measures both reflected and transmitted color, spectral reflectance, spectral transmittance and transmission haze. A host of measurement features and specialized sample-handling devices make UltraScan PRO the most versatile high-performance color measurement spectrophotometer available.

Fig. 8 UltraScan PRO

4 Results

4.1 Results of mechanical properties

4.1.1 Bending impact test

The bending impact test was measured according to EN ISO 179-1. The test bodies were set up horizontally on the supports and hammered by impact hammer strikes. The test bodies were 80x4x10 and were fitted with a notch type V at a depth of 2 mm. Twenty test bodies were used for each set of measurements.

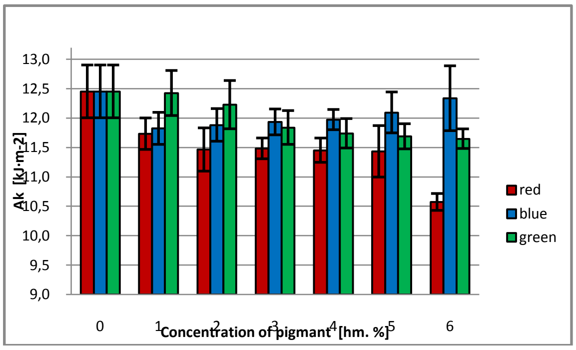

Fig. 9 Noch tougness

The bending impact test determines the stiffness of the test body. When adding a red (Lifocolor-CR-Red 3620 F/PC) (red) or green (Greocolor-CR-Green 4842 F/PC) natural green polymer pigment (lexan 923) a decrease in notched toughness can be observed with increasing pigment concentration. On the other hand, the addition of blue (Lifocolor -CR-Blue 50009 F / PC) (blue) pigment increased the notch toughness. It has also been shown that by adding the pigments, the toughness was reduced in comparison with the natural material.

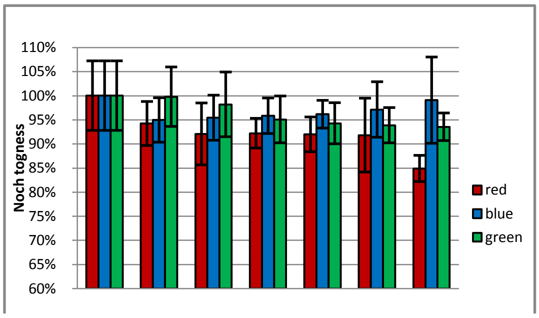

Fig. 10 Proportional graph of notch toughness

At 1% concentration of the red pigment there was a 6% decrease in notch toughness. Also a negative trend can be seen. As the concentration of red pigment increases, the impact strength decreases. At 6% concentration of the red pigment, the toughness decreases by up to 15%.

At 1% concentration of the green pigment, the impact strength value is the same as the value of the transparent material. At 6% concentration there was a 7% decrease in impact strength. The green pigment creates a more stable mixture with a decrease in the impact strength of the transparent material, and the stiffness of the test body has decreased.

At 1% concentration of the blue pigment, the toughness dropped by 5%. Further, the increasing trend is observed, at 6% concentration, the notched toughness value approaches the value of the transparent material. There was a 1% drop on the reference sample.

4.1.2 Tension test

The tensile test was performed according to the standard CSN EN ISO527-1 on the ZWICK Roell 1456 Testing Machine. The test bodies were shaped in the form of blades - type II. Twenty test specimens were used for each set of measurements. The temperature in the test room was 22 ° C.

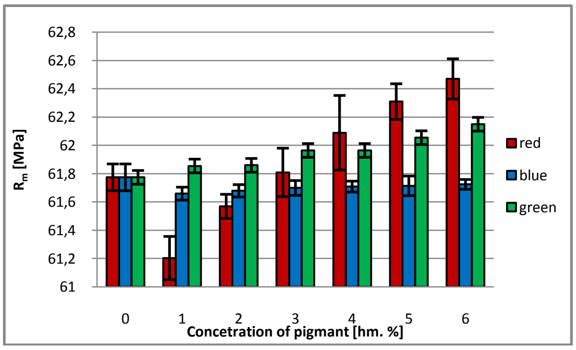

Fig. 11 Limit of strength

The tensile test indicates the strength of the material. When the red or green pigment was added to the natural polymer (lexan 923), the tensile strength was increased. On the other hand, when the blue pigment was added, a slight decrease in the tensile strength is observed.

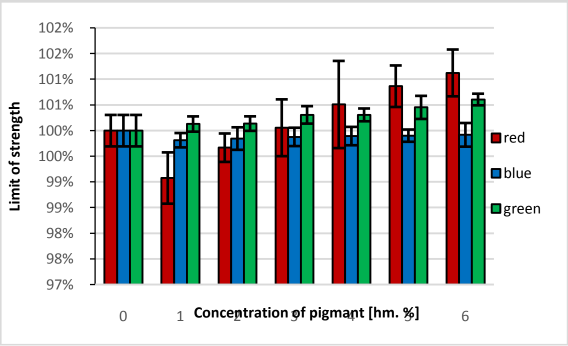

Fig. 12 Proportional graph Limit of strength

At 1% concentration of the red pigment, the strength limit was decreased by 1%, where a growing tren is evident. At 6% pigment concentration, the 1% strength limit was increased.

At 1% concentration of the green pigment, the strength limit was slightly increased. The 6% concentration increased by 0.6%.

In the blue pigment, the tensile strength of the tensile strength dropped. This value fluctuated within a range of 0.5%.

4.1.3 Shore D

The hardness test (Shore D) was performed according to ISO 7619-1 standard on the Omag Affri ART 13 hardness tester. The test bodies had a thickness at the test site of 8 mm and their surface was smooth and clean. Ten test bodies were used for each set of measurements and 10 measurements were performed. The temperature in the test room was 22 °C.

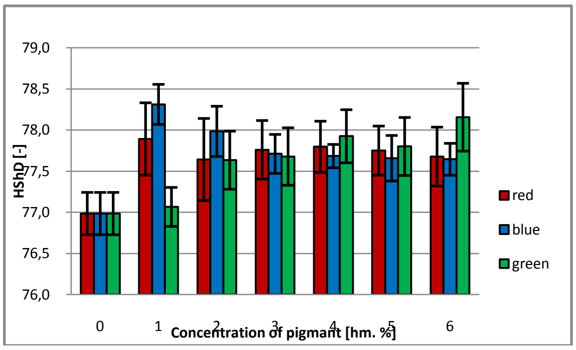

Fig. 13 Shore D

Hardness results (Shore D) show that when a red pigment was added to the PC material (Lexan 923), the hardness increased and the trend was decreasing with increasing concentrations. A similar phenomenon occurred with the use of a blue pigment. On the other hand, the green pigment showed a growing trend.

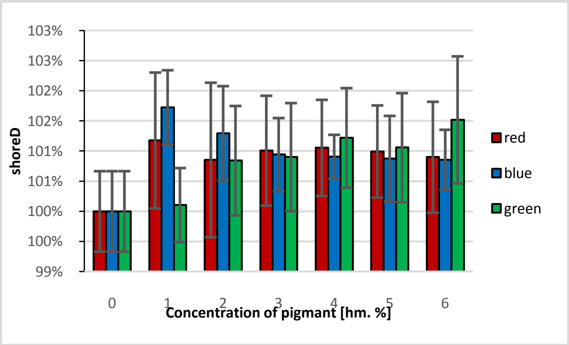

Fig. 14 Proportional graph ShoreD

The change of the hardness is the most notably with 1% blue pigment concentration. When concentration grew by 2%, the trend slightly declining. A similar phenomenon occurs when the red pigment is used. At 1% concentration the hardness increased by 1%. However, with the increasing concentration of the pigment, there was a slight decrease in hardness. The intense phenomenon occurred with the green pigment. At 1% concentration there was a slight increase in hardness. He was also watching the growing trend. At a concentration of 6%, the hardness increased by 2% with a transparent material

4.2 Results of colorimetric properties

To quantify the color shade of the respective concentration 5 test specimens were selected. These bodies were measured at ten different locations. These 50 measurements were subjected to statistical evaluation. The following colorimetric parameters are given as average values at the respective concentrations.

4.2.1 Red pigment

In the following figure can be seen that when a red pigment is used, it depends very much on the appropriate concentration in the mixture. Up to 3% of the pigment concentration in the mixture greatly changes the color gamut. Color variation is based on parameters a* and b*, often referred to as chroma C*.

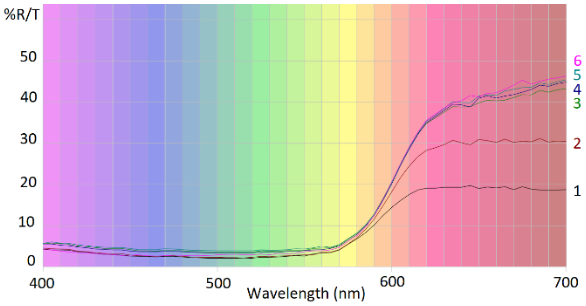

Fig. 15 Reflective spectrum

Increasing concentration of pigment in the mixture had a slight effect on variation in diversity. Above the 3% concentration slight shade changes also occurred. The hue is characterized by the L* parameter.

Table 3. Colorimetric parameters

	Red 1	Red 2	Red 3	Red 4	Red 5	Red 6
L*	29	33,8	36,02	35,46	37	36,5
a*	32,01	39,64	40,69	42,22	40,29	42,01
b*	17,55	22,62	21,8	23,23	20,47	26,63

4.2.1 Green pigment

Using a green pigment, the samples were already stained with the desired variety at 1% concentration, but the high homogeneity of the mixture was not guaranteed. This was manifested by large interquartile dispersion from the reflection curve

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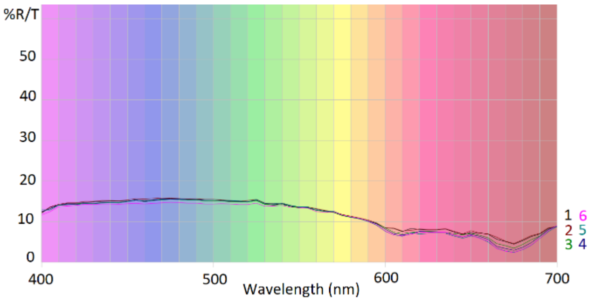

Fig. 16 Reflective spectrum

At 3% concentration there has been a significant reduction in scattering. And again the shade value changed, but the human eye almost invisible.

Table 4. Colorimetric parameters

	Green1	Green2	Green3	Green4	Green5	Green6
L*	41,6	41,95	41,98	42,07	42,25	42,56
a*	-10,88	-12,31	-11,72	-10,91	-10,25	-10,02
b*	-5,58	-6,04	-5,98	-6,04	-6,19	-6,13

4.2.1 Blue pigment

At 3% concentration there has been a marked reduction in scattering. And again, the shade value has changed, but the human eye is almost unrecognizable.

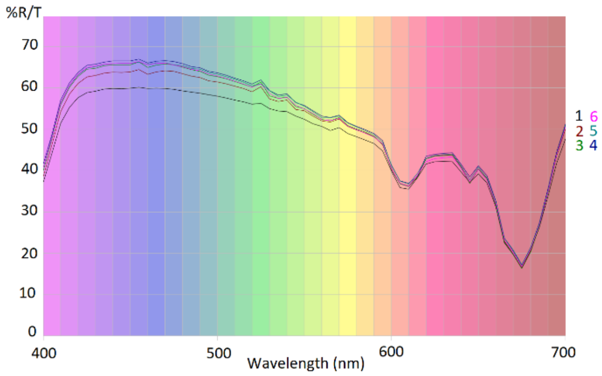

Fig. 17 Reflective spectrum

Above 3%, no shade changes occurred.

Table 5. Colorimetric parameters

	blue 1	blue 2	blue 3	blue 4	blue 5	blue 6
L*	76,76	77,99	78,37	78,47	78,52	78,67
a*	-8,99	-9,65	-9,9	-9,88	-9,89	-9,88
b*	-8,12	-9,38	-10,3	-10,15	-9,97	-10,43

5 Conclusion

Impact strength evaluation shows that the type of pigment selected significantly influences the stiffness of the body. When red pigments were used at higher concentrations, the stiffness decreased up to 15%. The blue pigment influenced the stiffness of the material to a minimum.

The tensile test evaluation shows that pigments do not significantly affect the tensile strength. Selection of the right concentration will result in minimal deviations from the strength limit.

Hardness is also influenced but only on a small scale. We are in the area of minimum deviations when a 3% pigment concentration is used. At this concentration, the materials have very similar mechanical properties.

At lower concentrations there is a great variation in diversity. At a concentration of 3%, blends achieve optimal coloring, both in color and shade. Increasing concentration above 3% no longer plays a significant role.

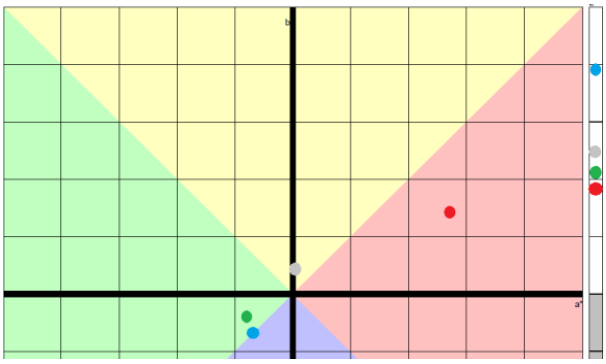

Fig. 18 3% Pigment concentration

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